Status report on Beamline 10.3.2

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Beamline 10.3.2 has had a varied history, starting as an optics-development beamline on which new techniques could be explored, then evolving into an X-ray microprobe beamline. In its latest incarnation, it's a hard X-ray microprobe beamline optimized for environmental studies, with μEXAFS and μSXRF capabilities. Two years ago, the optics were changed to allow more flux at the cost of a bigger, but adjustable, spot size. The beamline came up to full user operation in November, 2001 and is now fully booked. While most of the users have been looking at speciation of metals in earth materials, there have also been projects in archeometry, osteology and microbiology.

The beamline can deliver white or monochromatic beam in a spot ranging from 16x6μm (full flux) down to 5x4μm FWHM. The energy range is 3-17keV. The optical layout consists of a horizontally deflecting 1:1 toroid, focusing to a slit, followed by optics inside the hutch, consisting of a vertically collimating parabolic mirror, 4 crystal monochromator, horizontally focusing ellipse and a vertically refocusing parabolic mirror. The optical layout is shown in Figure 1.

The sample is carried on a an XY stage with 10nm steps, and fluorescence radiation is detected by a 7-element Ge detector. Fluorescence mapping is done in a continuous-scan fashion in which the stage moves at a constant speed. The detector electronics allows one to define spectral regions of interest (ROIs) in which counts are accumulated. At fixed distance intervals, these counts are loaded into successive locations in the internal memory of the detector electronics. Thus, a line scan is built up without the need to step and repeat for each point. Successive line scans result in a two-dimensional fluorescence map.

These maps may be used to choose points at which to do EXAFS. This combination of fluorescence mapping and EXAFS is very useful in deciphering the structures of complex, inhomogeneous systems. Also, we have recently acquired a CCD for powder diffraction. Once that is integrated into the system, we will have the uniquely powerful combination of μSXRF, μEXAFS and μXRD, all available on the same sample in the same run.

An example of a fluorescence map from Alain Manceau's work is shown in Figure 2. This illustration shows gray-scale maps of the same ferromanganese soil nodule taken in the 'light' of several different elements. One can see, for instance, that areas high in Mn are low in Fe, that Ca exists mostly in the form of isolated mineral grains, and that As is found where Fe is. Such information, backed up with detailed analysis of EXAFS spectra, can shed light on how trace elements are sequestered in the environment, and hence add scientific rationale to the process of soil remediation.

For the April shutdown, we are planning to replace the 4-bounce monochromator with a 2-bounce, constant-height unit, which will yield a 3-10x flux improvement. Also, we plan to implement automatic mirror tuning, which will result in smaller spots and greater reliability. We now also have a Bruker SMART 6000 diffraction camera for the end station, and this will be fitted by summer '02. This will allow a range of diffraction experiments to be conducted, and will offer a similar capability to beamline 7.3.3, but with the addition of XAS. We are also examining the improvement in performance that would result from moving the system to a superbend. Improvements range from around 10 at 12 keV to nearly 100 at 20 keV, and would allow a much wider range of elements to be studied.

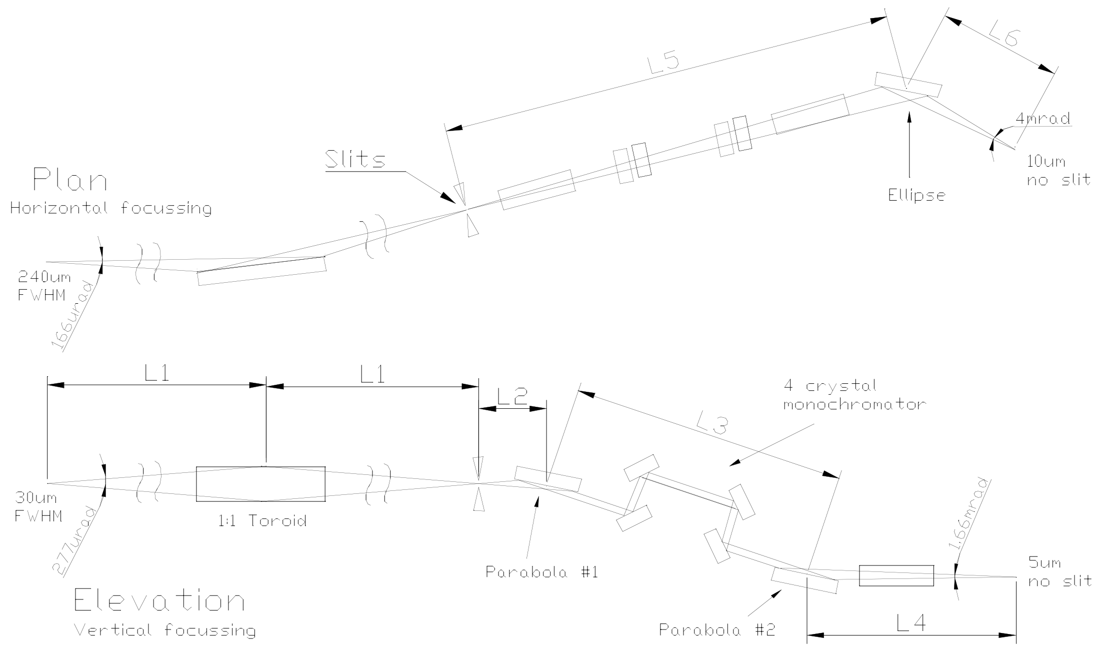

Figure 1. Optical layout. The dimensions L1..L6 and mirror types are as follows:

L1 = 14.75m
L2 = 1.44m
L3 = 0.820m
L4 = 0.26m
L5 = 2.4m
L6 = 0.12m

Mirror	*Type*	*Active area*
M1	*1:1 toroid*	*612 x 4.63mm*
M2	*V parabola*	*100 x 0.23mm*
M3	*V parabola*	*100 x 0.37mm*
M4	*H ellipse*	*100 x 0.66 mm*

Mirror grazing angles = 4 mrads (coating = 8nm Rh on 20nm Pt on 5nm Cr)

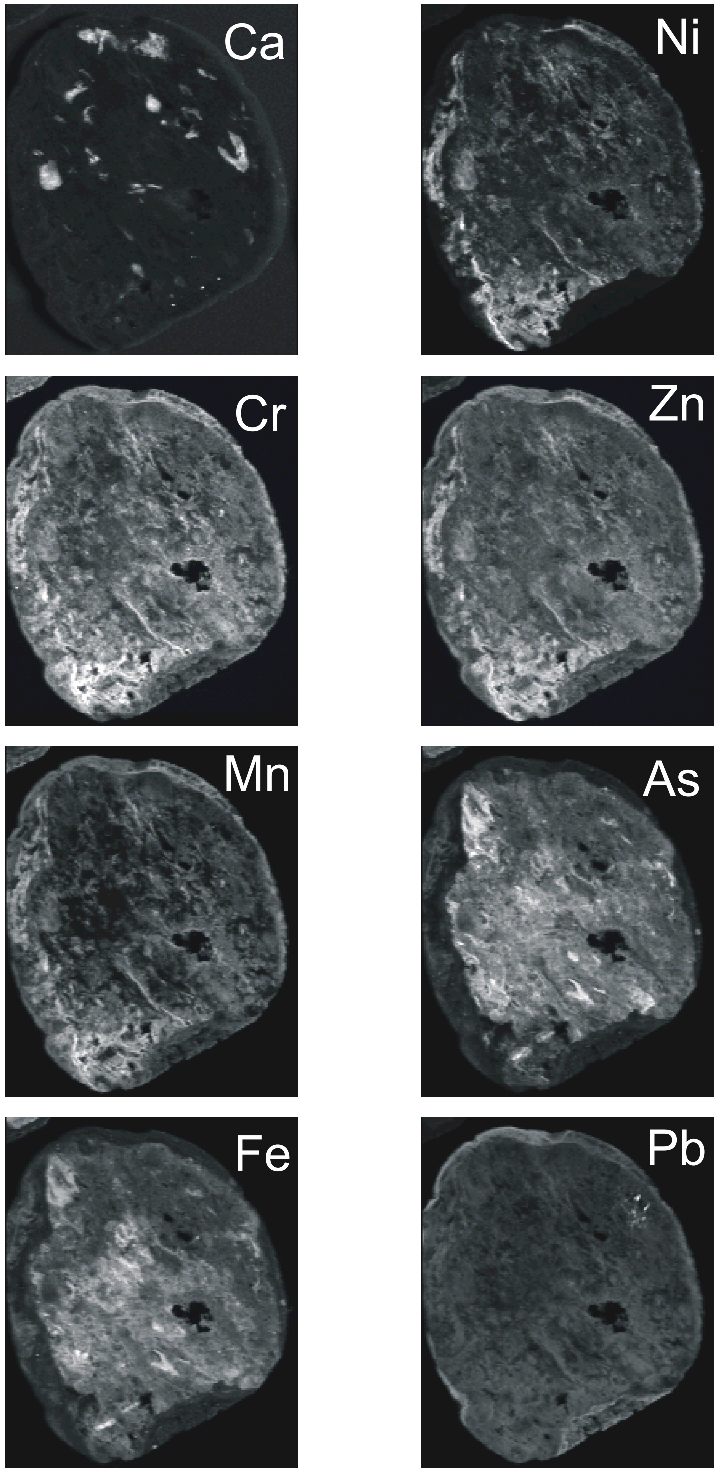

Figure 2. Elemental maps of a ferromanganese nodule. The As and Pb maps were taken below and above the Pb L_{III} edge, respectively, in order to separate the signals from these two elements.

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